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S. W. STRATTON, DIRECTOR

No. 446

[Part of Vol. 18]

SPECTROPHOTOELECTRICAL SENSITIVITY OF ARGENTITE (Ag_2S)

BY

W. W. COBLENTZ, Physicist

Bureau of Standards

AUGUST 18, 1922

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SPECTROPHOTOELECTRICAL SENSITIVITY OF ARGENTITE (Ag_2S).

By W. W. Coblentz.

ABSTRACT.

The present paper, considered in connection with some previously published data, represents a study of the effect of crystal structure upon photoelectrical sensitivity, as observed in silver sulphide, Ag_2S, in the isometric form, argentite, and in the orthorhombic form, acanthite.

Experimental data are given on the effect of temperature, of the intensity of the radiation stimulus, and of mechanical working of the material, upon the spectrophotoelectrical sensitivity of argentite. These observations are then compared with similar data, previously published, on acanthite.

The spectrophotoelectrical reactions of these two minerals were found to be quite unlike, from which it is concluded that crystal structure has a marked effect upon photoelectrical sensitivity.

CONTENTS.

I. INTRODUCTORY STATEMENT.

The present paper gives the results of a continuation of previous work on the spectrophotoelectrical properties of various substances,[1] the chief point of interest being the effect of crystal structure upon photosensitivity.

[1] General Survey (Bismuthinite), B. S. Sci. Papers, No. 322; Molybdenite, B. S. Sci. Papers, No. 338; Silver Sulphide, B. S. Sci. Papers, No. 344; Thalofide, B. S. Sci. Papers, No. 380; Positive and Negative Conductivity Phenomena, B. S. Sci. Papers, No. 398; and Proustite, B. S. Sci. Papers, No. 412.

As the general survey of the spectrophotoelectrical conduction in solids progresses, it appears more and more evident that the phenomenon is very complex. Several general characteristics of photoelectrical response appear to be in common with all the substances [2] thus far investigated, and can be formulated.[3] In addition to these general properties, it appears that each photosensitive substance, thus far examined, has a characteristic spectral response, just as each fluorescence substance has a characteristic spectrum.

Methods of experimentation can not as yet be formulated, and in order to find some clue to the explanation of the phenomenon of photoelectrical conduction, experimenters have been groping along searching for relations between other optical phenomena (as for example, transmission, reflection, etc.); also applying various tests, as, for example, heat and mechanical treatment, effect of temperature, and intensity of the radiation stimulus.

The early experiments were made chiefly upon selenium, which in the form of a grid seems too complex for study. Each newly discovered photosensitive substance seems to yield new results. For example, some substances (molybdenite, stibnite, etc.) show that the photosensitivity is localized in spots. In bismuthinite the photosensitivity was found most marked along the intergrowth of two bundles of acicular crystals. In the samples of silver sulphide (in the form of the mineral acanthite) examined electrolytic polarization did not appear to affect the induced photoelectrical conduction. In certain samples of molybdenite,[4] radiation stimuli of certain wave lengths induce a counter emf. Evidently if all these reactions underlie the general phenomenon of photo-electrical conduction in solids, the whole is very complex.

Included in the aforementioned list of photoelectrical reactions is the effect of crystal structure. Brown[5] has given data on the physical properties of selenium crystals formed by sublimation in an electric oven. The spectrophotoelectrical sensitivity curves of samples formed at high temperatures (210° C.) are different from those of crystal masses formed at lower temperatures.

Silver sulphide, Ag_2S, as a mineral, occurs in two forms; *argentite* which is isometric and *acanthite* which is orthorhombic. Both minerals were found by Case[6] to be photoelectrically sensitive and

[2] Including unpublished data on bournonite, pyrargyrite, etc.
[3] B. S. Sci. Papers, 16, p. 635, 1920; Jr. Op. Soc. Am., 4, p. 249, 1920.
[4] Including unpublished data.
[5] Brown, Phys. Rev. (2), 4, p. 85; 1914.
[6] Case, Phys. Rev. (2), 9, p. 305; 1917.

hence form interesting material for studying the effect of crystal structure upon photosensitivity.[7]

Data on the spectrophotoelectrical sensitivity of acanthite were given in a previous paper,[8] in which it was shown that (for all wave lengths) the radiation stimulus appears to induce a photonegative response (increase in resistance, or counter emf) which is superposed upon the photopositive response usually observed in substances exhibiting photosensitivity. Moreover, at 20° C., the photoelectrical reaction is high for radiations of wave lengths 0.6 to 1.1μ, followed by an unsymmetrical maximum at 1.4μ. At low temperatures (−150 °C.) the sensitivity curve is symmetrical with the maximum shifted to 1.2μ. The general outline of the curve exhibiting the spectrophotoelectrical reaction differs greatly for different samples. Since the samples examined are usually aggregates of crystals, which are no doubt irregularly arranged, it would appear that, in the orthorhombic crystal, the inequality of the axes has a marked effect upon photoelectrical conduction.

In the present paper it will be shown that argentite differs from acanthite in that the radiation stimulus does not induce a photonegative reaction. Moreover, the spectrophotoelectrical reaction for all samples, at room temperatures, is confined practically to a narrow spectral band, with a maximum at 1.35μ, instead of being spread out over a wide spectral region as obtains in acanthite. At low temperatures the sensitivity curve remains narrow, with the maximum shifted to 1.1μ.

II. APPARATUS AND MATERIAL.

For making the observations in the infra red and the visible spectrum the spectroradiometric apparatus consisted of a mirror spectrometer, quartz prism, and a 500-watt gas-filled tungsten lamp described in previous papers.[9] In the visible spectrum the data were supplemented with measurements made with a lens spectrometer and a glass prism.

The radiation intensities ($E=1$, $E=6.4$, etc., see Fig. 2) were applied in the form of an equal energy spectrum by varying the current through the lamp, as in previous work.

The measurements in the ultra violet (0.3μ to 0.4μ) were made with a quartz lens spectropyrheliometer,[9] the source of radiation

[7] Coblentz, Amer. Phys. Soc., February, 1922, Phys. Rev, (2) 19, p. 532, 1922.
[8] B. S. Sci. Papers, 15, p. 231; 1919.
[9] B. S. Sci. Papers, 15, p. 120, 1919; 17, p. 180, 1921; (spectropyrheliometer), 16, p. 233, 1920.

being a Cooper-Hewitt 600-watt quartz-mercury lamp. The emission lines of mercury were reduced to equal-energy stimuli (in terms of $\lambda = 0.334\mu$ which was used at its full intensity) by suitably diaphragming the quartz lens which was used to focus an image of the burner upon the spectrometer slit. In this manner high intensities were obtained which permitted an extension of the photoelectrical measurements into the ultra violet.

The material used came from Freiberg, Saxony, Germany. Samples Nos. 1, 2, 4, 6, and 7 were broken from a large, quite well-formed crystal. Sample No. 5 was a well-formed crystal, broken from a long branched formation—United States National Museum, specimen No. 78474. Sample No. 3 was broken from a large massive formation of small crystals showing cleavage surfaces 0.5 mm. or smaller in diameter.

Grooves were cut around the ends of these samples into which was wound fine, No. 36, copper wire. This wire and the ends of the crystals were then completely covered with Woods alloy, which provided good electrical contact over the whole end of the crystal.

The dimensions between these metal electrodes were as follows: No. 1, 3 by 2 by 1.5 mm; No. 2, 5.5 by 2.5 by 1.5; No. 3, 6 by 7 by 4; No. 4, was hammered flat, 4 by 2.7 by 0.2 mm; No. 5, 8 by 4 by 3 mm.; No. 6, was cut thin, 3.5 by 2.5 by 0.5; No. 7, 6.5 by 2.5 by 2.5 mm (one side rubbed flat).

In these tests the photoelectrically sensitive substance was connected directly in circuit with a d'Arsonval galvanometer, a high resistance and a suitable potential obtained from "dry batteries." The usual procedure was to apply sufficient potential to obtain a suitable galvanometer deflection. At room temperature 2.6 volts sufficed, but at low temperatures the resistance was so high that 50 to 80 volts were required to obtain the desired photoelectric deflections.

In general, it was desired to map the relative spectral photoelectric responses for a given temperature, and hence no attempt was made to obtain the data at the same intrinsic sensitivity which increases with decrease in temperature. In order to reduce the observations to the same intrinsic sensitivity it would have been necessary to use the same "dark current" at all temperatures. However, in studying the effect of intensity of the radiation stimulus upon the spectrophotoelectrical reaction, both the temperature and the applied voltage (to give the same dark current) remained constant.

III. EXPERIMENTAL DATA.

Under this caption illustrated data are given, indicating the progress of the photoelectrical reaction of argentite with time of exposure to radiation; also the effect of temperature, intensity of the radiation stimulus, etc., upon the photoelectrical conductivity. These data are then compared with similar data previously published on acanthite.

1. EFFECT OF DIRECTION OF CURRENT.

In previous investigations on molybdenite it was shown that in samples exhibiting a photonegative reaction, this phenomenon was selective to the wave length of the radiation stimulus, and depended upon the direction of the current through the crystal.

On the other hand in acanthite it was found that the photonegative effect was produced by all wave lengths of the radiation stimulus, and it did not depend upon the direction of the electric current through the crystal. Furthermore, the tests at room temperature and at low temperatures appeared to indicate that the photoelectrical reaction does not depend upon any polarization effects inherent in the crystal.

In the present investigation it was found that at room temperature and at $-100°$ C., in argentite, like acanthite, the dark conductivity is greater in one direction than in the reversed direction through the crystal. At both high and low temperatures the induced photoelectric currents were correspondingly different. That is to say, if the dark current on reversal ("current reversed," *C. R.*) was one-half that observed for the current direct (*C. D.*), then the corresponding photoelectrical response (galvanometer deflection) was reduced to one-half that observed for the "current direct." From this it appears that the unipolar conduction inherent in argentite affects the magnitude of the dark current through the crystal but it does not appear to affect the spectrophotoelectrical reaction.

2. RESPONSE-TIME CURVES.

Under this caption it will be shown that the increase in electrical conductivity with time of exposure of argentite to radiation and the recovery after exposure are entirely different from that of acanthite.

In previous papers [10] it was shown that at room temperature the resistance change in acanthite when exposed to radiation

[10] B. S. Sci. Papers, 15, p. 236, 1919; 16, p. 604, 1920.

is the result of a photopositive and a photonegative reaction. The photopositive response (decrease in resistance) appears to be complete in a few seconds. The photonegative response builds up slowly and disappears slowly, as is evident from the negative galvanometer deflection after removing the light stimulus. At low temperatures the photonegative reaction disappears and 90 per cent of the photopositive reaction occurs during the first few seconds, while equilbibrium is attained in the course of 3 to 5 minutes. Furthermore, the time for recovery is the same as the

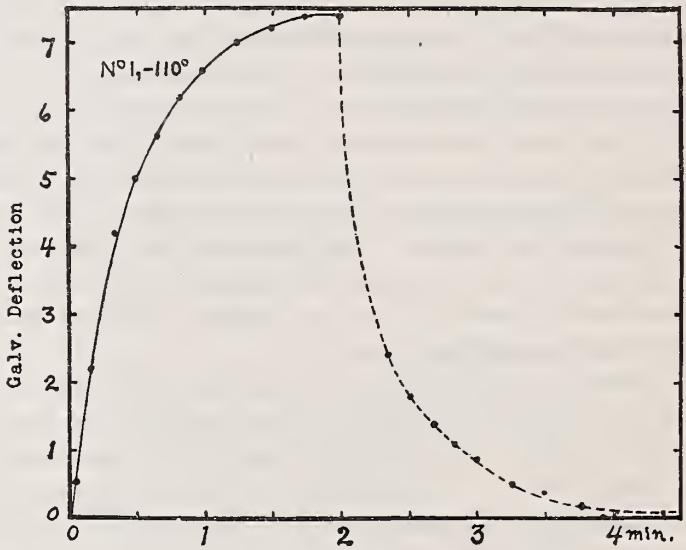

FIG. 1.—*Reaction-time curve of argentite.*

time of response, namely, 3 to 5 minutes. The results of the present investigation show that in argentite the photopositive reaction at room temperatures is practically instantaneous just as observed in acanthite; but there is no building up of a photonegative reaction which delays the time of attaining equilibrium observed in acanthite. Hence, at room temperature the maximum of the galvanometer deflection for all wave lengths of the applied radiation stimulus was attained in 3 seconds, which was the time of swing of the galvanometer coil. As the temperature was decreased the time to attain the maximum of the photoelectric reaction was gradually prolonged from 10 to 30 seconds.

At the lowest temperatures attained the time required to obtain the maximum of the photoelectrical response was prolonged to 3 or more minutes, with an equally long time for recovery (dotted part of curve in Fig. 1). This is illustrated in Figure

1, in which, however, the exposure was terminated at the end of 2 minutes, though it is evident that equilibrium had not yet been established. A comparison of this curve with that of acanthite[11] shows that the rate of increase in the photoelectrical reaction in argentite is not so rapid as in acanthite.

Just what bearing these data will have on the general subject of photoelectrical reactions in solid conductors can not be foretold. Suffice it to say that they establish a distinct difference in the photoelectrical reactions of the two crystal forms of silver sulphide, one (the orthorhombic) of which shows a photonegative

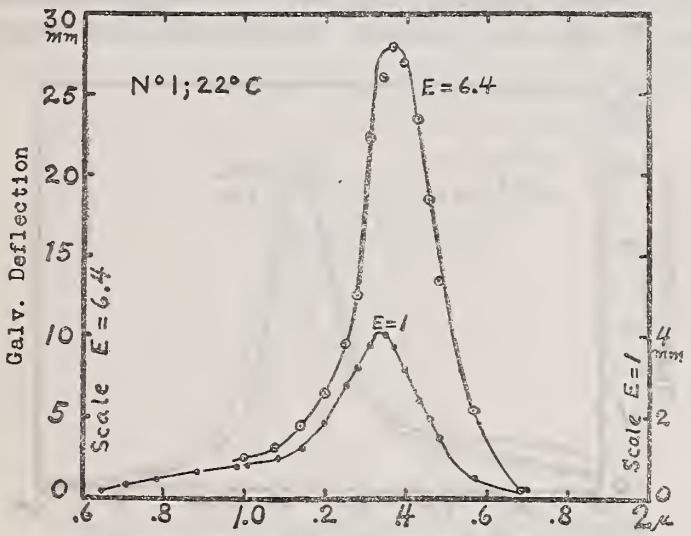

FIG. 2.—*Effect of intensity of radiation upon the spectrophotoelectrical reaction of argentite.*

reaction while the other (the isometric) does not exhibit this property.

3. EFFECT OF THE INTENSITY OF RADIATION STIMULUS UPON THE ELECTRICAL CONDUCTIVITY.

In Figure 2 is shown the effect produced upon the spectrophotoelectrical sensitivity of argentite on varying the intensity of the radiation stimulus. Increasing the intensity 6.4 times has a marked effect in increasing the photoelectrical sensitivity on the long wave length side of the maximum as previous observed in acanthite and molybdenite. For example, at 1μ the ratio of the galvanometer deflections for $E = 1$ and $E = 6.4$ is 3, while for $\lambda = 1.36\mu$ this ratio is 7. This is practically the same as observed in

[11] B. S. Sci. Papers, 15, p. 237, Fig. 5.

acanthite. This ratio increases with wave length, being = 10 at 1.57μ.

The conclusion to be drawn from these observations is that a variation of the intensity of the radiation stimulus produces the same spectrophotoelectrical reaction in these two crystal forms of silver sulphide.

4. PHOTOELECTRICAL SENSITIVITY OF DIFFERENT SAMPLES.

In the previous paper on silver sulphide, it was shown that different samples, (also different parts of the same sample) of acanthite exhibit markedly different spectrophotoelectrical reactions. In all cases, however, the photoelectrical sensitivity,

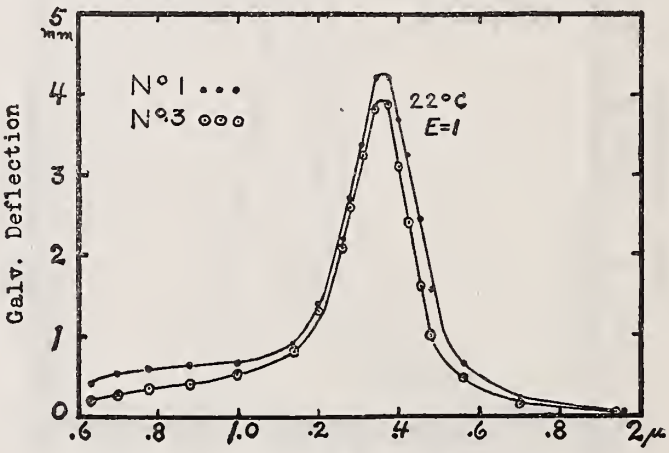

FIG. 3.—*Comparison of different samples of argentite.*

at room temperature, was found to be high throughout the visible spectrum, to 1.1μ in the infra-red, followed by an unsymmetrical maximum at about 1.38μ.

An examination of the illustrations presented in the present paper shows a conspicuous similarity of the spectrophotoelectrical curves of the 7 samples of argentite examined. Throughout the visible spectrum to 1.1μ in the infra-red the photoelectrical sensitivity is low, followed by a symmetrical maximum at 1.33μ to 1.35μ.

This marked similarity of the spectrophotoelectrical sensitivity curves of different samples of argentite is illustrated in Figure 3. As already described, sample No. 1 was cut from a single well-formed crystal, while No. 3 was broken from a massive block consisting of microscopic crystals.

From these data and those previously published on acanthite, it appears that crystal structure has a marked effect upon spectrophotoelectrical sensitivity.

5. EFFECT OF TEMPERATURE UPON SPECTROPHOTOELECTRICAL SENSITIVITY.

It was shown in the previous paper on acanthite that the polarization phenomenon which was observed at room temperatures disappeared at low temperatures. Moreover, the unsymmetrical spectrophotoelectrical sensitivity curve became symmetrical, with a maximum at 1.2μ, at low temperatures ($-160°$ C.).

In Figure 4 is shown the effect of temperature upon the spectrophotoelectrical reaction of argentite, sample No. 1. The maximum

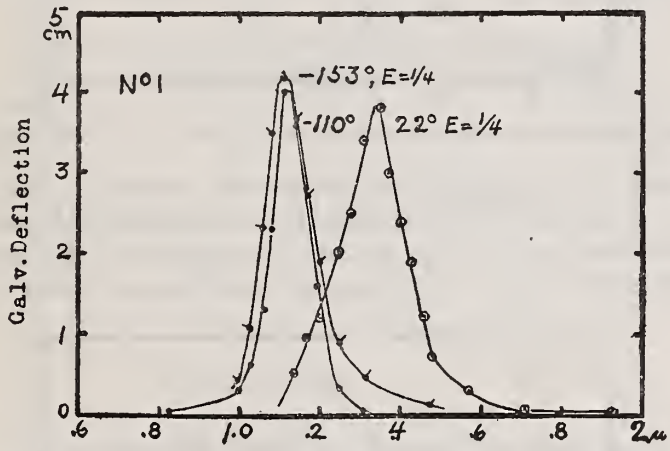

FIG. 4.—*Effect of temperature upon the photoelectrical reaction of argentite.*

sensitivity shifts from 1.34μ at 22° C. to 1.12μ at $-110°$ C., and to 1.11μ at $-153°$ C. In Figure 4 the galvanometer deflections at 22° C. are in millimeters and the intensity of the radiation stimulus, $E=1/4$ (as compared with $E=1$, etc., in Figure 2), was the same at all temperatures. In view of the fact that the main interest appeared to be to obtain a relative comparison of the spectrophotoelectrical sensitivity curves, no attempt was made to vary the voltage so that, at all temperatures, there would be the same dark current. Usually the radiation intensity was the same at all temperatures, except in cases where, at room temperature, the intensity was not sufficient to observe the maximum accurately. The lowest intensity, $E=1/4$, permitted observations over a wider spectral range (using an equal energy spectrum) than was possible with the higher intensity.

In Figure 5 is shown the effect of temperature upon the spectrophotoelectrical sensitivity of sample No. 2. Similarly in Figure 6 is shown the spectral reactions of sample No. 3, which, as already

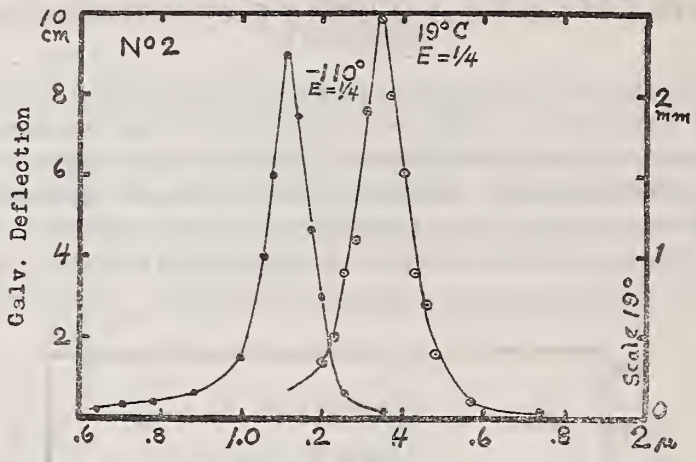

FIG. 5.—*Effect of temperature upon the photoelectrical sensitivity of argentite.* (Sample 2.)

mentioned, is an aggregate of microscopic crystals. The maximum, at −145° C., does not appear to be shifted quite so far to the short wave lengths (to 1.13μ) as in some of the other samples. In Figure 6 the scale of ordinates is in millimeters for 21° C. and

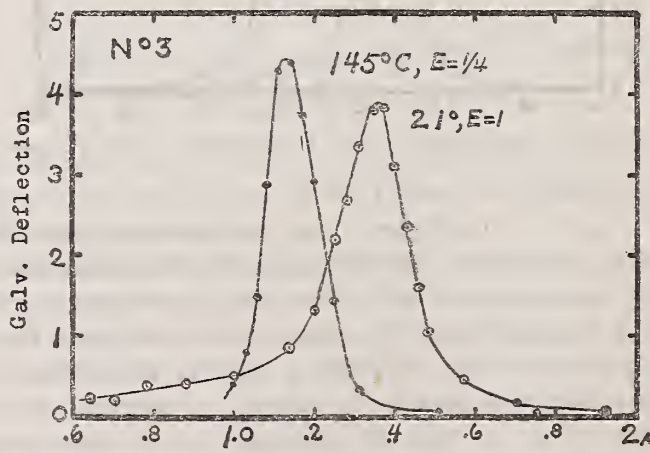

FIG. 6.—*Effect of temperature upon the photoelectrical sensitivity of argentite.* (Sample 3.)

for centimeters −146° C. The spectrophotoelectrical reaction of sample No. 5 (U. S. National Museum specimen No. 78474) is illustrated in Figure 7. At 24° C. the maximum occurs at 1.36μ, which may be owing to the high intensity used. At −137° C. the maximum is shifted to 1.07μ, which is somewhat shorter than the

average wave length (1.11μ) for the other samples at the lowest temperature attained.

In Figure 8 is illustrated the effect of temperature upon the spectrophotoelectrical sensitivity of a polished surface of sample No. 6. As will be noticed under the following caption, the fact that the maximum occurs at 1.33μ at a temperature of 22° C., and at 1.11μ at −141° C. indicates that the mechanical polishing of the surface does not appear to affect the spectrophotoelectrical sensitivity.

6. EFFECT OF MECHANICAL WORKING UPON SPECTROPHOTOELECTRICAL SENSITIVITY.

In view of the fact that the samples of argentite examined could not be severed from the original crystal without cutting

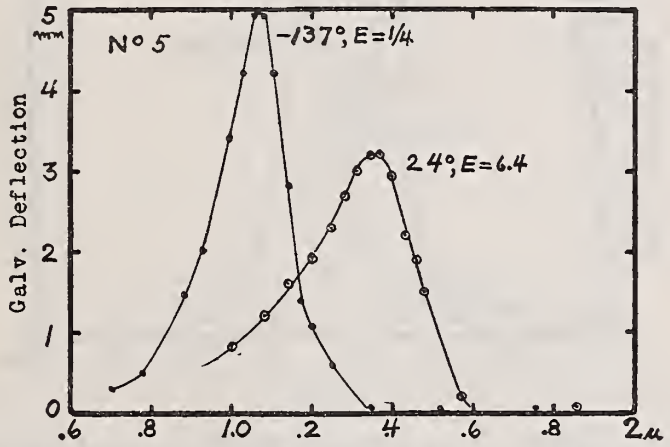

FIG. 7.—*Effect of temperature upon the photoelectrical sensitivity of argentite.* (Sample 5.)

and fracturing, it was of interest to determine whether mechanical working affected the spectrophotoelectrical reaction. The previous investigation showed that mechanical working of acanthite greatly affected its photoelectrical reaction.

In Figure 9, curves *A* and *B* give the spectrophotoelectrical sensitivity curves of two rough crystalline cleavage surfaces of argentite sample No. 7. The two curves *C*., for 22° C. and −114° C., give the spectrophotoelectrical reactions of another surface of this same sample which had been rubbed and polished on a file to such an extent that some of the material flowed over the edge. The maximum at 1.34μ for 22° C. (and 1.1μ for −114° C.), as well as the general outline of the sensitivity curve, does not differ from that of the crystal surfaces, showing that this superficial

working has no marked effect upon the spectrophotoelectrical sensitivity.

In Figure 10 the circles (⊙ ⊙ ⊙) illustrate the spectrophotoelectrical sensitivity of sample No. 6 cut thin and flat with a knife. Similarly the dots and commas (. . . and , , ,) show the reaction of the opposite side which was filed flat and polished. The effect of superficial working is not more marked than that caused by the small variations in photosensitivity observed in different samples.

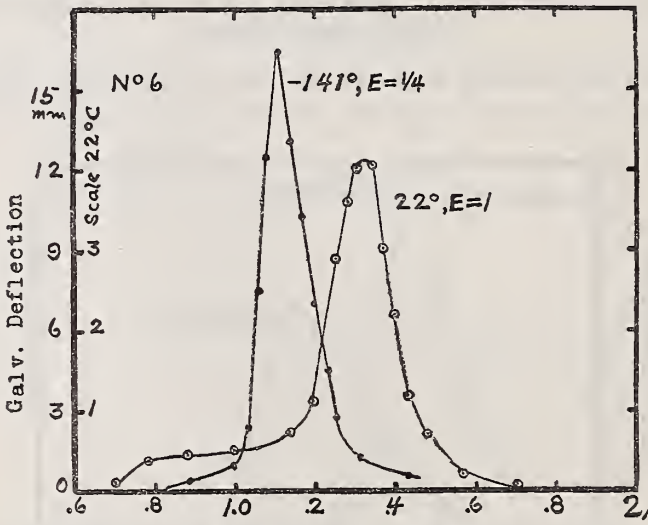

FIG. 8.—*Effect of temperature upon the photoelectrical sensitivity of argentite.* (Sample 6.)

Argentite sample No. 4 was hammered thin and presented a smooth flat surface. As shown in Figure 11, at 25° C. the maximum of photosensitivity, at 1.34μ, is not affected by mechanical working. However, at −175° C. the maximum is shifted but little toward the short wave lengths, being at 1.2μ instead of 1.1μ as observed on the unwrought samples. This is in exact agreement with the data obtained on samples of acanthite, which had been subjected to a similar mechanical treatment. A further observation of interest is that at −175° C. the time of exposure required to obtain a maximum response was only 10 to 30 seconds as compared with 1 to 2 minutes for the unworked samples.

No doubt in this hammered, pliable, condition the crystal structure of these two minerals has been greatly changed, and perhaps reduced to a microcrystalline state. Hence a similarity in their photoelectrical properties should, no doubt, be expected.

But the chemical composition has not been affected, and silver sulphide, Ag_2S, gives a characteristic photoelectrical response spectrum, just as obtains in other substances (e. g., selenium, stibnite, and cuprous oxide) which react photoelectrically.

7. PHOTOELECTRICAL SENSITIVITY OF SILVER SULPHIDE IN THE ULTRA VIOLET.

Using the quartz spectropyrheliometer mentioned on a previous page, the ultra violet spectrophotoelectrical sensitivity of argentite and acanthite was determined at room temperature. The data obtained are to be considered preliminary to a more thorough

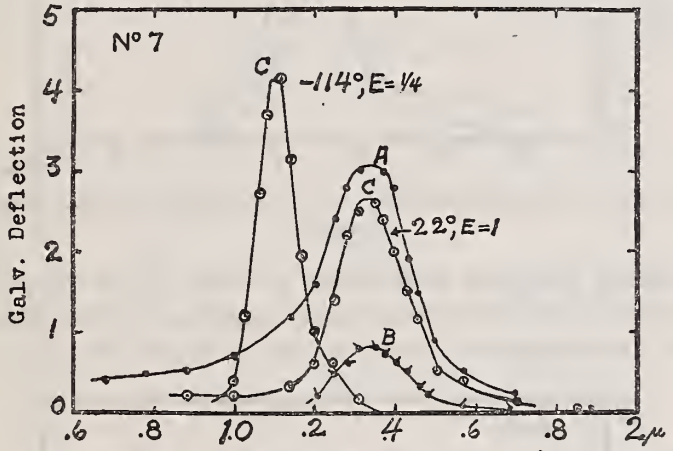

FIG. 9.—*Effect of mechanical working upon the photoelectrical reaction of argentite.* (Sample 7.)

examination with apparatus better adapted for investigations in the ultra violet.

The measurements were extended to 0.25μ, and the results obtained are interesting in showing that silver sulphide is photoelectrically sensitive to radiations extending throughout this spectral region. Exact measurements were made using the mercury lines 0.305μ, 0.313μ, 0.334μ, 0.365μ, 0.405μ, 0.436μ, and 0.546μ, reduced to the same radiometric intensity.

In Figure 12 the dotted curves illustrate the photoelectrical measurements on argentite sample No. 1, and acanthite sample No. 6, in the visible spectrum, the source of radiation being a 600-watt stereopticon gas-filled tungsten lamp. These observations verify previous results, showing that acanthite is more sensitive than argentite in the spectral region from 0.6μ to 1μ thus producing an assymmetrical maximum in the infra red.

The ultra violet spectrophotoelectrical sensitivity of argentite sample No. 1, and of acanthite sample No. 6 is illustrated with the continuous curves in Figure 12, the source of radiation being an

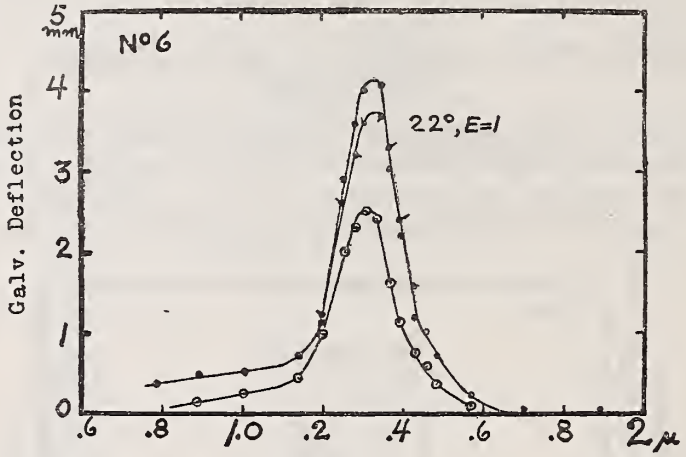

FIG. 10.—*Effect of mechanical working upon the photoelectrical reaction of argentite.* (Sample 6.)

equal energy spectrum of a quartz mercury vapor lamp already mentioned. The data indicate high sensitivity in the ultra violet with an unsymmetrical maximum in the region of 0.41μ.

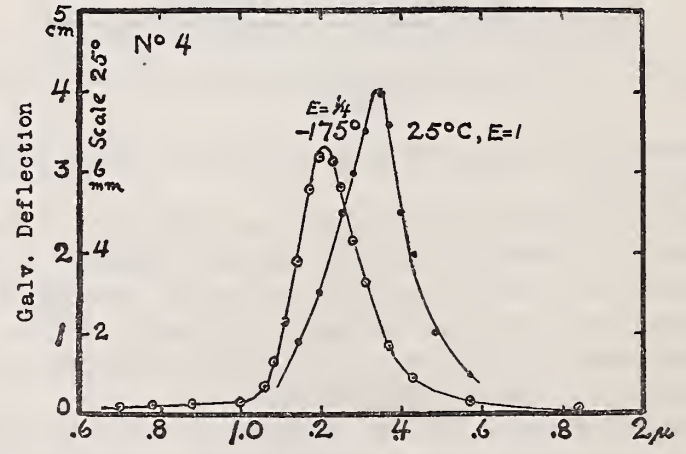

FIG. 11.—*Effect of mechanical working upon the photoelectrical reaction of argentite.* (Sample 4.)

In view of the different spectral apparatus employed, no exact comparison can be given of the photoelectrical reactions in the ultra violet relative to the infra red. The observed ultra violet sensitivity was considerably less than that of the infra red.

The light gathering power of the spectropyrheliometer was twice that of the mirror spectrometer. The intensity of the ultra violet radiation stimulus was therefore not less than $E = 1$ (see Fig. 2), and it may have been greater than $E = 6.4$. It is therefore a conservative estimate to say that the sensitivity maximum in the infra red is from 3 to 10 times greater than the one in the ultra violet.

The general results obtained are interesting in showing that the spectrophotoelectrical sensitivity of silver sulphide consists of two unsymmetrical maxima, one in the ultra violet and the other in infra red. Whether this has any significance in the generalization of photosensitivity, when considered with the fact that many of

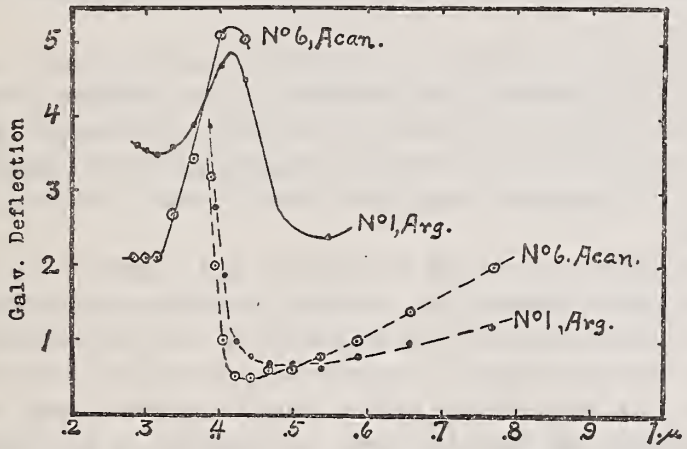

FIG. 12.—*Photoelectrical sensitivity of acanthite and argentite in the visible and ultra violet spectrum.*

the photoelectrically sensitive solids, thus far examined, have two or more maxima remains to be determined.

IV. SUMMARY.

In the foregoing pages data are given on the change in electrical resistance of silver sulphide, in the form of the mineral argentite, when exposed to radiations of equal energy and of wave lengths extending from 0.3μ in the ultra-violet spectrum to 2μ in the infra-red, beyond which wave length this mineral appears to be photoelectrically insensitive.

The effect of temperature, of the intensity of the radiation stimulus, and of mechanical working upon spectrophotoelectrical sensitivity of argentite (isometric crystal) was investigated, and the observations compared with similar data, previously published, upon acanthite (rhombohedral crystal).

Argentite reacts slightly, photoelectrically, to radiations of wave lengths 0.5 to 1.1μ, followed by a sharp fairly symmetrical maximum at 1.35μ. In contrast with this is acanthite, which reacts quite strongly to radiations at 0.5 to 1.1μ, followed by an unsymmetrical maximum at 1.35μ. Both minerals have a high photoelectrical reaction in the ultra-violet with maximum in the region of 0.41μ.

At low temperatures the intrinsic photoelectrical sensitivity of argentite is greatly increased and the maximum shifts to the short wave lengths, — to 1.1μ as compared with 1.2 for acanthite under similar conditions.

The photoelectrical reaction of argentite differs from that of acanthite in being free from an induced photonegative polarization.

In argentite, as previously observed in acanthite (also molybdenite, etc.), increasing the intensity of the radiation stimulus produces a more rapid reaction in the long wave lengths than in the short wave lengths, with a consequent shift of the maximum of the photoelectrical sensitivity curve toward the long wave lengths.

Hammering the crystals of argentite and acanthite into thin, pliable plates, lowers the intrinsic photoelectrical sensitivity. The position of the infra red maximum of spectrophotoelectrical sensitivity of the worked samples is less affected, by changing the temperature, than obtains in the natural crystalline state; and it is practically the same for these two minerals, at low temperatures. From this it appears that aside from the effect of crystal structure, silver sulphide has a characteristic photoelectrical response spectrum.

From a comparison of the spectrophotoelectrical reactions of these two crystal forms of silver sulphide under various conditions, it appears permissible to conclude that crystal structure has a marked effect upon spectrophotoelectrical sensitivity.

WASHINGTON, April 8, 1922.